

Review

Additively Manufactured Ni–Co Superalloys for Hydrogen Safety Enhancement of Gas-Turbine Energy Systems: Microstructural Degradation and Crack Initiation Mechanisms

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Abstract

Ni–Co γ/γ' -strengthened superalloys are key structural materials for modern energy and flow turbomachinery systems due to their exceptional high-temperature strength, creep resistance, as well as hydrogen and corrosion stability. However, operation in gaseous hydrogen environments typical of hydrogen-cooled generators, cooled gas-turbine blades, and emerging hydrogen-energy technologies can significantly affect their microstructural stability and fracture behavior. This study presents a comprehensive multiscale review of hydrogen-induced nanoscale degradation and crack initiation mechanisms in Ni–Co superalloys produced by wrought, powder metallurgy, and additive manufacturing routes. Transmission electron microscopy combined with quantitative morphometric analysis was employed to characterize the size, morphology, and spatial distribution of γ' precipitates, revealing a dense population of coherent particles predominantly in the 40–120 nm range, governed by a log-normal distribution. Correlations between precipitate size, aspect ratio, and circularity indicate the onset of partial loss of coherency and coarsening for particles exceeding ~80 nm, creating favorable sites for hydrogen localization. The presence of TCP phases (η , σ , μ , Laves) and carbides at grain boundaries and within grains was shown to enhance microstructural heterogeneity and act as effective hydrogen traps, promoting interfacial decohesion and microcrack initiation. To support microstructural interpretation, convolutional neural network analysis with Grad-CAM visualization was applied to SEM images, enabling the identification of the structural regions most sensitive to hydrogen-assisted damage, particularly γ/γ' interfaces and defect clusters. The results demonstrate that hydrogen-induced degradation in Ni–Co superalloys is governed by the coupled interactions among microstructure, hydrogen distribution, and local stress state. The findings provide a physically grounded basis for optimizing alloy chemistry, heat treatment, and additive manufacturing parameters, as well as for developing AI-assisted predictive models for the durability of critical components in hydrogen-energy and high-temperature power-generation systems to increase hydrogen safety.

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Keywords: Ni–Co superalloys; γ/γ' phases; hydrogen embrittlement; nanoscale degradation; microstructure; MC/M₂₃C₆ carbides; TCP phases (η , σ); TEM/SEM analysis; Grad-CAM; additive manufacturing; hydrogen charging; crack initiation; additive manufacturing; 3D printing

1. Introduction

Ni–Co superalloys are widely used in power-generation systems, aerospace engineering [1–3], and turbomachinery due to their high thermal stability, corrosion resistance [4–6], and ability to retain their mechanical properties under long-term loading in high-temperature environments [6–8].

However, operation in hydrogen-containing conditions—particularly in hydrogen-cooled turbogenerators [9,10] and gas-turbine engine blades—may lead to hydrogen absorption, which significantly affects the microstructure and durability of these steels and alloys [11–14]. Studies have demonstrated that hydrogen can penetrate along grain boundaries, induce local stress accumulation, and initiate microcracks, especially in regions with structural heterogeneity [1,15–18]. This results in reduced fracture toughness, an increased tendency toward brittle failure, and degradation of properties in defect-rich zones. Therefore, understanding the effects of hydrogen charging on fracture toughness, durability, and hardness is crucial for predicting the remaining service life of Ni–Co superalloy components operating in aggressive hydrogen-containing environments.

2. Current State of Heat-Resistant Gas-Turbine Ni–Co Materials

Ni–Co superalloys possess exceptionally high melting temperatures (up to ~90% of T_m), enabling their use in the hot sections of aerospace gas-turbine engines, including turbine blades, working discs, rings, and combustion chambers. Owing to their excellent resistance to creep, corrosion, and oxidation at temperatures above 700 °C, these alloys ensure increased efficiency and long-term durability of high-temperature systems [19–22]. These properties determine their widespread use as structural materials for manufacturing critical components of thermal power systems, aerospace engines [23–26], reactor equipment, steam turbine components, heat exchangers, and other technologies, where the combination of thermal stability, strength, and long-term durability is essential [27–29]. Based on the production method, superalloys are classified as cast, deformed, powder, and additively manufactured superalloys.

Cast nickel–cobalt superalloys are multiphase alloys strengthened with intermetallics and carbides, designed to provide exceptionally high mechanical strength, thermal stability, and corrosion resistance at temperatures up to ~1000 °C [29–32]. Their compositions are optimized through controlled ratios of Ni and Co, while the addition of Cr, W, Al, Ti, Nb, and Hf promotes the stabilization of the γ' phase and the formation of a protective Al₂O₃ oxide layer [33]. This oxide film significantly enhances high-temperature strength and corrosion resistance [34–36]. In particular, the newly developed cast high-chromium Co–Ni-based alloy 9CoNiCr demonstrates the following performance characteristics [33]: a thermally stable γ' microstructure at 900–1000 °C with significantly slowed γ' -phase coarsening; the formation of a continuous protective Al₂O₃ layer, providing oxidation resistance comparable to that of advanced Ni-based superalloys such as CMSX-4; a high yield strength and a competitive minimum creep rate at 950 °C. Studies of the Co/Ni ratio in γ' -strengthened alloys (ERBOCo-1/1X) have demonstrated that an optimized elemental balance provides superior high-temperature strength, driven by modifications in γ' morphology and reduced dislocation energy [34,37]. Cast Ni–Co–Cr–W–Mo systems stabilize the γ' phase and topologically close-packed (TCP) phases in accordance with service con-

ditions, which has been confirmed through detailed investigations of phase composition and TCP clustering behavior [35]. Ultimately, optimized cast superalloys exhibit exceptional high-temperature strength, long-term operational capability under severe thermal loads, and enhanced oxidation and corrosion resistance, making them well suited for aerospace turbine components, power-generation units, and nuclear systems [36,37].

Wrought (cast/forged) Ni–Co superalloys containing balanced proportions of Ni and Co exhibit improved mechanical performance, including high yield strength, impact toughness, and enhanced creep resistance even at temperatures up to ~950 °C. Plastic deformation processes—such as forging or cold rolling—produce fine-grained structures, high dislocation densities, and nanoscale γ' precipitates; these features strengthen the alloy by increasing the yield strength (up to ~1400 MPa at 923 K) and suppressing grain growth during service-related heating [38].

Studies of cold-rolled and annealed Ni–25Co–Al–Ti–Cr–Mo–W alloys revealed a trimodal γ/γ' microstructure with nano-dispersed γ' lattices and hundreds of nanotwins, resulting in exceptional strength even at 1023 K (1086 MPa) [38]. Hot-compression experiments demonstrated that refined grain formation and the development of subgrain-scale nanograins through micro-twinning significantly modify the deformation mechanisms within the strain range of 0.1–1.0, thereby optimizing the strength–ductility balance [39,40].

The Co/Ni ratio is critical for stabilizing the γ' phase and improving creep resistance at 900 °C (as shown in single-phase single-crystal studies), demonstrating the key role of the balance between the principal alloying elements in forming a thermally stable microstructure under prolonged high-temperature exposure [41–44]. Such wrought Ni–Co alloys are widely employed in aerospace turbines, power-generation systems, and other applications where ultrahigh strength, structural stability, and resistance to deformation at elevated temperatures are essential [45–48].

Powder metallurgy, like casting, allows products of a specified shape to be produced without significant mechanical processing, and deformation [49–52]. After hot isostatic pressing, the parts are free of liquation [53], micropores and structural heterogeneity [54–56]. They have a fine-grained microstructure with many refractory elements and a uniform distribution of strengthening phases. This structure ensures phase stability, high short-term, long-term, and fatigue strength over a wide temperature range (up to 1100 °C), durability, and crack resistance, particularly in gaseous hydrogen [57].

Selective laser melting (SLM) and directed energy deposition (DED) methods are used for additive manufacturing of nickel-based alloys [58–61]. The prospects and economic efficiency of additive manufacturing for producing complex parts for the energy, aerospace, and automotive industries are demonstrated. The structures formed during powder melting processes have been analyzed, and γ , γ' , γ'' , TCP phases, carbides, nitrides, oxides, and borides have been identified, depending on their composition [58]. To reduce microstructural heterogeneity, a method of continuous laser forming with in situ heat treatment (IHT) has been developed for homogenization of AM DD98 m samples [62]. A comprehensive review of in situ alloying technology in laser additive manufacturing has revealed the key advantages of this method, namely flexibility in the selection of chemical composition and reduced production costs, as well as significant problems such as chemical heterogeneity, unmelted particles, and structural defects [63,64]. This study emphasizes the need for precise control of laser melting parameters and post-processing conditions to ensure a homogeneous microstructure and stable mechanical properties.

Regardless of the production technology, the microstructure features of heat-resistant Ni–Co superalloys determine their operational characteristics [65–68]. It has been established that the quantity, type, size, and morphology of intermetallic and carbides determine the functional characteristics of modifications of the Inconel 718 nickel alloy

with different chemical compositions and production technologies (casting, wrought metallurgy, powder metallurgy, and additive manufacturing), temperature, homogenization duration, and aging [69–72]. The heat resistance of alloys depends on structural heterogeneity [53–56], the strength of interatomic bonds in a solid solution [65,67], the type, quantity, dispersion, morphology, distribution, and thermal stability of strengthening phases [65–67], grain size and grain boundary condition [52,53,67], diffusion process parameters, content of modifying elements and harmful impurities [52,53,67].

One of the key features of Ni–Co superalloys is the dependence of the morphology and distribution of the γ' phase on the Ni/Co ratio. At high Ni content, γ' precipitates tend to adopt a spherical morphology, whereas at higher Co content, they become cubic [57,68]. This behavior is attributed to changes in the γ/γ' lattice mismatch caused by the distribution of W between the phases, which also affects the creep behavior at 900 °C. According to [57], increasing the Co content from 13% to 35% leads to a reduction in the average grain size (from ~26 μm to ~18 μm), as well as an increase in the size of the “nano-phase” precipitates (from ~80 to ~230 nm). A comprehensive analysis of the distribution of the γ' phase, carbides, and the σ phase was developed, which is essential for understanding variations in mechanical strength and high-temperature stability. In multicomponent Ni–Co alloys, MC carbides, M₂B borides, and potentially the σ phase are often found along grain boundaries. EBSD–EDS analysis has shown that in Co/Ni superalloys these phases form due to local enrichment in W and Mo. Their presence influences grain-boundary properties and the overall stability of the microstructure [68]. Alloying up to 2 at.% Re in the Co–30Ni–10Al–5Mo–2Nb superalloy changes the morphology of γ' from cuboidal to rounded cuboidal due to a decrease in the crystal lattice mismatch (from ~+0.32% to ~+0.19%) [69]. This modification also increases the stability of the γ/γ' interface during prolonged annealing at 900 °C. All the structural features mentioned above and their evolution during operation affect the sensitivity of nickel–cobalt alloys to hydrogen-containing environments [71–74].

Therefore, the aim of the work is to investigate the mechanisms of hydrogen-induced nanoscale degradation and crack initiation in γ/γ' -strengthened Ni–Co superalloys by establishing the relationship between structural components, precipitate morphology, and hydrogen localization, using modern microscopy methods and AI-assisted image analysis to contribute to enhancing the hydrogen safety of modern energy systems.

3. Materials and Methods for Investigation

3.1. Investigated Alloys

Ni–Co heat-resistant superalloys of the Ni₅₆Cr₁₄Co₁₅Mo₅Al₃Ti₃ (EP-741NP) type are used for manufacturing discs of modern gas-turbine engines (GTEs) and gas-turbine units (GTUs) for land- and marine-based applications, including systems operating on alternative fuels. This study examined the influence of hydrogen on the properties of the heat-resistant nickel-based powder superalloy Ni₅₁Cr₉Mo₃W₆Nb₂AlTi (KhN51KVMTYB) (EP-741P) and the wrought vacuum-induction-melted superalloy Ni₆₂Cr₁₄Mo₅Nb₃AlTi (KhN62BMKTYu) (EP-742) (Firth Rixson Metals Ltd., Glossop, UK), whose chemical compositions and mechanical properties are presented in [1].

3.2. Microscopy Imaging

Microstructural examinations were performed using a Hitachi SU-70 field-emission scanning electron microscope (Hitachi High-Tech Corporation, Tokyo, Japan) equipped with secondary-electron (SE) and backscattered-electron (BSE) detectors. Imaging was conducted at an accelerating voltage of 30 kV, a working distance of 5.4 mm, and in TE mode.

Thin foils for the microstructural examination of the Ni–Co superalloy were prepared according to standard transmission electron microscopy specimen preparation procedures. Samples were first sectioned from the working zone of the alloy and mechanically ground to a thickness of ~80–100 μm . Final thinning was performed by electropolishing in a two-jet electrolyte holder at low temperature (–20 to –30 $^{\circ}\text{C}$) until an electron-transparent region (“hole with a rim”) was obtained. In selected cases, Ar^+ ion milling was used for final surface cleaning to remove the mechanically damaged layer. TEM foils were mechanically ground to approximately 100 μm , punched into 3 mm discs and twin-jet electropolished in a solution of 10 vol.% perchloric acid and 90 vol.% ethanol at –30 $^{\circ}\text{C}$ until perforation occurred. Specimens for cross-sectional transmission electron microscopy (TEM) (using HR-TEM-Fei Techai G2 F20 S Twin, FEI Company, Hillsboro, OR, USA) were prepared using a standard TEM sample preparation approach, including cutting, grinding, and dimpling. Ar ion milling was carried out at a voltage of 4.0 kV with an angle of 6° , followed by a final low-voltage ion-milling of 2.5 kV with an angle of 2° as a final step. The electron-transparent regions adjacent to the perforation were used for TEM examinations. Fractographic investigations were performed using an EVO-40XVP electron microscope (Carl Zeiss SMT AG, Oberkochen, Germany) equipped with an INCA Energy 350 microanalysis system (Oxford Instruments Plc, Abingdon, UK). The microstructure was investigated by Zeiss Stemi 2000-C Stereo Microscopes (Carl Zeiss AG, Oberkochen, Germany) and SIGETA industrial color digital camera (UCMOS 1300, 1.3 MP and SIGETA International Color Digital Camera MCMOS 5100 5.1 MP) (SIGETA, Hong Kong, China). The image size of all TEM micrographs recorded on a CCD camera was kept at 2004 pixel $\times$ 1335 pixel. The exposure time for the HRTEM images was set to 1.0 s. The aberration coefficients were set to be sufficiently small for obtaining the HRTEM images under slightly over-focus conditions (close to Scherzer defocus). The strain field in CrN/AlN multilayer was calculated on the CS-corrected HRTEM (JEOL 2100F, JEOL Ltd., Tokyo, Japan) and STEM images (FEI Titan Themis 60-300 X-FEG S/TEM instrument operated at 300 kV, equipped with a probe corrector for spherical aberration, FEI Company, Hillsboro, OR, USA) by geometric phase analysis (GPA) method.

Observations were carried out using a transmission electron microscope in bright-field (BF), dark-field (DF), and selected-area electron diffraction (SAED) modes. The accelerating voltage was set to 200–300 kV, providing sufficient penetration and contrast for examining γ/γ' phases, dislocation structures, and hydrogen-induced nanopores. Images were captured using a high-resolution digital camera. All focusing parameters, apertures, and objective-lens settings were optimized to minimize artifacts and ensure maximal clarity in the regions exhibiting local degradation.

To improve the accuracy of the analysis, the images were additionally processed using standard TEM procedures, including contrast adjustment, noise reduction, and scale calibration. SAED patterns were employed to refine the phase composition and determine the orientation relationships of the γ/γ' phases. All TEM data were correlated with the results of SEM analysis and microhardness measurements to obtain an integrated assessment of hydrogen-induced degradation in the material.

3.3. Statistical Processing of Microstructural Parameters

The statistical analysis of the morphometric parameters of γ' precipitates in the Ni–Co superalloy was performed using TEM micrographs (200 nm scale) acquired in standard bright-field mode. Initial image processing was conducted in the ImageJ 1.53k/Fiji environment using built-in modules such as Thresholding, Analyze Particles, and filters based on area and shape. Prior to segmentation, the images were converted to 8 bit format, and either the automatic Otsu threshold or a manually adjusted threshold was applied to isolate the γ' phase as dark regions against the γ -matrix background.

After binarization, morphological cleaning of the mask (de-speckle, remove outliers, binary opening) was performed to minimize the influence of noise and artifacts.

Particles larger than 10 nm were selected for further analysis, excluding partially cut objects located at the image boundaries. For each precipitate, the equivalent diameter, area, perimeter, circularity ($4\pi A/P^2$), and aspect ratio (major/minor axes) were determined. The complete set of morphometric data was exported in .csv format and analyzed in Python 3.12.6 using the NumPy 2.1.1, SciPy 1.14.1, Matplotlib 3.9.2, and Pandas libraries 2.2.3.

The particle-size distribution was approximated using log-normal and gamma models, for which the parameters μ , σ , shape k , and scale θ were determined. Quantitative descriptors included the mean value, D_{50} (the median), D_{10} , and D_{90} , the standard deviation, the geometric mean, and the variance. Additionally, correlations between particle size and aspect ratio, as well as the circularity–size relationship, were evaluated using Pearson’s correlation coefficient r . To assess spatial homogeneity, a nearest-neighbor distance (NND) distribution was constructed.

A total of ≈ 300 γ' precipitates were analyzed for the statistical dataset, ensuring statistically representative results. The obtained morphometric characteristics were used to assess the growth behavior of the γ' phase, the degree of coalescence, and the evolution of the microstructure.

3.4. Grad-CAM Heatmap Construction

The implementation of the research methodology was based on earlier studies [73,74]. To interpret the behavior of the convolutional neural network and identify the microstructural regions that most strongly influence the model’s decision, the Gradient-weighted Class Activation Mapping (Grad-CAM) method was applied. SEM images were first normalized, resized to match the model input dimensions, and fed into the CNN. The activation tensors of the last convolutional layer and the corresponding gradients of the model output with respect to these activations were then extracted and stored.

The importance weights were calculated by globally averaging the gradients, after which a weighted sum of the activation maps was constructed. A ReLU function was applied to the resulting map to retain only the positive contributions that increase the probability of the target class. The final Grad-CAM heatmap was interpolated to match the size of the original SEM image and overlaid on it as a semi-transparent color layer.

The resulting heatmaps make it possible to identify local microstructural features (pores, γ' precipitates, γ/γ' heterogeneities, defective zones) that the model considers most informative. This provides a visually interpretable explanation of the CNN’s decision-making process and enhances the reliability of automated microstructure analysis in Ni–Co superalloys. Recent advances in artificial intelligence (AI) and machine learning have significantly expanded the capabilities of microstructural characterization in materials science. Convolutional neural networks (CNNs) have been successfully applied to scanning and transmission electron microscopy images for automated phase identification, defect detection, microstructure classification, and prediction of mechanical properties. In studies of hydrogen-related degradation, AI-assisted image analysis enables the extraction of complex microstructural information that may be difficult to quantify using conventional approaches. Furthermore, explainable AI methods, such as gradient-weighted class activation mapping (Grad-CAM), provide visual interpretation of the regions that most strongly influence model predictions, thereby increasing the transparency and reliability of deep learning-based materials diagnostics. Therefore, the application of CNN-based and Grad-CAM techniques represents a promising approach for the analysis of microstructural evolution and hydrogen-induced damage in Ni–Co superalloys.

3.5. Hydrogen Charging of the Ni–Co Super Alloys

A set of research instruments for studying the influence of hydrogen and hydrogen-containing gases on the properties of structural materials has been developed [1–3]. The purity of the hydrogen atmosphere has a significant effect on the degree of hydrogen embrittlement in structural metallic materials [75–78]. Therefore, the technical-grade hydrogen was additionally purified and dried, and all tests in gaseous hydrogen were carried out according to the following procedure [79–82]:

The testing chamber was evacuated to 0.13 Pa after placing the specimens inside.

The chamber was then filled with hydrogen, purged, and evacuated again.

Hydrogen was subsequently introduced to the required pressure, and the tests were performed.

After this procedure, the hydrogen volume fraction in the chamber reached 99.9997%, the oxygen volume fraction did not exceed 0.00007%, and the mass concentration of water vapor was 0.0009 g/m³. The hydrogen content was determined by the vacuum-fusion method using a LECO TCH 600 analyzer [83], and the measurement results are reported in [1].

4. Results and Discussion

4.1. Microstructural Examination and Analysis of the Ni–Co Superalloys

The microstructure of the Ni–Co superalloys was examined (Figure 1a). The image reveals a fine-dispersed phase structure with dark, rounded contrast features—typical of γ' precipitates ($\text{Ni}_3(\text{Al}, \text{Ti})$) embedded in the γ matrix (a Ni–Co–Cr solid solution). The observed contrast is most likely caused by differences in chemical composition or coherency stresses between the γ and γ' phases.

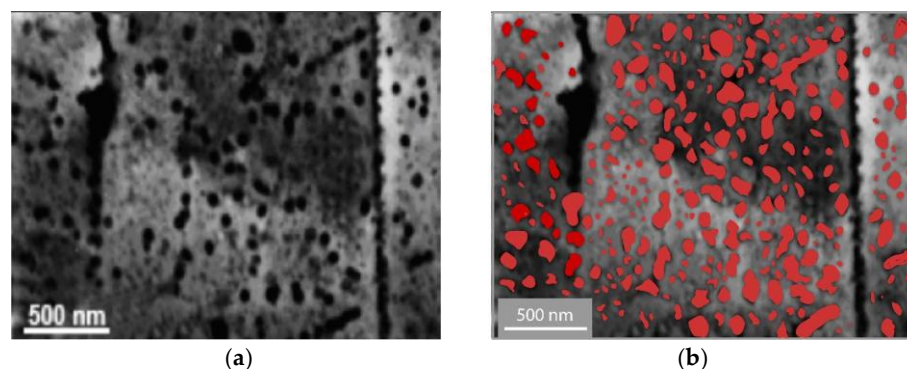


Figure 1. A fragment of the Ni–Co superalloy microstructure obtained by transmission electron microscopy (TEM) in bright-field mode (a). Preparation of the image for statistical analysis—highlighting the structural constituents, the γ' phase, in red (b).

The main structural constituents are as follows: dark particles with diameters of 10–50 nm, corresponding to coherent γ' precipitates oriented according to the cubic lattice of the γ matrix; bright regions corresponding to the γ matrix (a solid solution of Ni, Co, Cr with W, Mo, Al, and Ti); occasional linear contrasts (a vertical trace on the right and a crack or sub-boundary on the left)—likely representing deformation bands or subgrain boundaries. Local clusters of precipitates, indicating possible aging heterogeneity or partial γ' -phase coarsening. Such a microstructure is characteristic of thermally strengthened superalloys, such as the Inconel series and EP742 alloys, or Ni–Co–Cr–Mo–Ti–Al systems after aging at 700–800 °C [81–84]. The high density of fine precipitates provides dislocation strengthening and enhances creep resistance. The histogram shown in Figure 2 illustrates the particle-size distribution (in nanometers). Most precipitates fall within the 40–120 nm

range, which is typical of secondary γ' phases in nickel-based superalloys such as Inconel 718 or EP-742. A smaller number of larger particles (up to 300 nm) indicates aggregation or partial γ' -phase coarsening during aging. The pink curve represents the fitted normal distribution. Most inclusions are concentrated in the 60–100 nm range, which is characteristic of secondary γ' -phase particles in Ni–Co-based superalloys. A statistical analysis of this type of alloy is presented in [69,85].

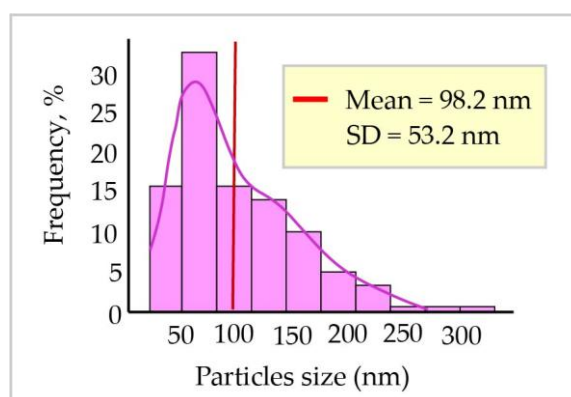


Figure 2. Histogram of the size distribution of precipitates (structural constituents— γ' phase) with a fitted normal distribution (Gaussian) curve. Number of precipitates: 282. Standard deviation: ≈ 53.2 nm. Mean size: ≈ 98.9 nm.

Table 1 and Figures 3–6 present and visualize the further statistical analysis of the structural constituents— γ' -phase precipitates. The microstructure provides an effective strengthening mechanism by impeding dislocation motion through fine γ' precipitates. This indicates a high potential of the materials for application in turbomachinery components operating at elevated temperatures.

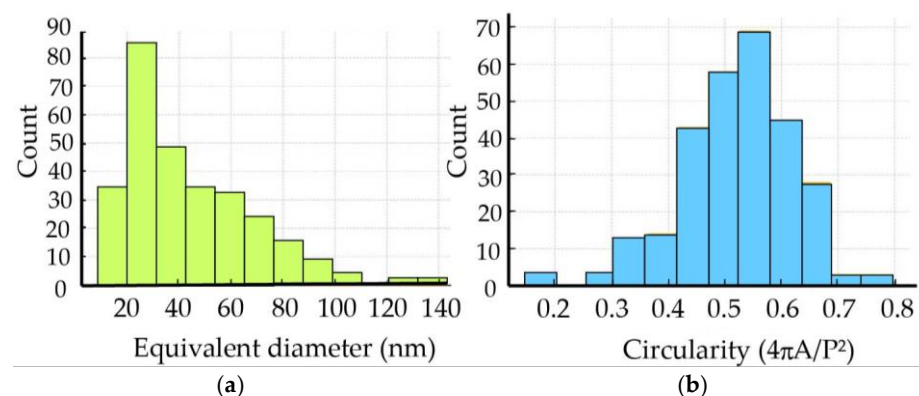


Figure 3. Distribution of the equivalent diameter of the γ' -phase precipitates (a). Distribution of the circularity of the γ' -phase precipitates (b).

Table 1. Size distribution of γ' precipitates. The size ranges represent equivalent particle diameters (nm) determined from image analysis.

No	Precipitate Size Range (nm)	Count	Fraction (%)
1.	21–60	76	27.0
2.	60–99	91	32.3
3.	99–138	52	18.4
4.	138–177	37	13.1
5.	177–216	15	5.3

The histogram in Figure 4a shows that most precipitates have an aspect ratio (major/minor) in the range of 1.3–1.7, indicating a morphology close to spherical. Only a small fraction of the particles are elongated (aspect ratio > 2). This reflects the predominance of coherent and uniform γ' -phase growth, which is characteristic of nickel-based superalloys after optimal heat treatment. Figure 4b shows a statistical distribution with a maximum in the 50–80 nm range, confirming the high density of γ' precipitates and the effectiveness of their dispersion strengthening through dislocation-motion hindrance. A long tail is observed on the right side of the histogram, which may be associated with local aging heterogeneity.

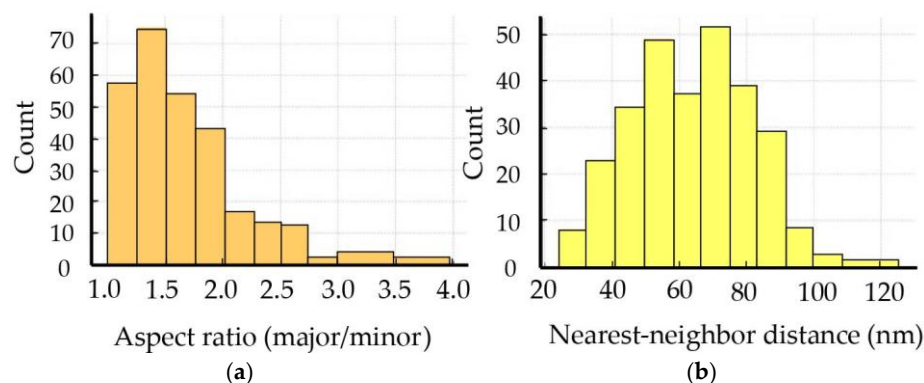


Figure 4. Aspect ratio (a). Nearest-neighbor distance distribution for the γ' -phase precipitates (b).

The log-normal fitting results (Figure 5a) confirm the classical nucleation and growth mechanism of the γ' phase, without any explosive (abnormal) particle coarsening. This is consistent with the precipitation behavior typical of Ti/Al-strengthened Ni–Co superalloys.

The CDF curve (Figure 5b) shows that:

- ~50% of the particles have $D < 80$ nm;
- ~90% have $D < 120$ nm.

Thus, the secondary γ' phases dominate, and their morphology remains stable under conditions typical of high-temperature turbine operation.

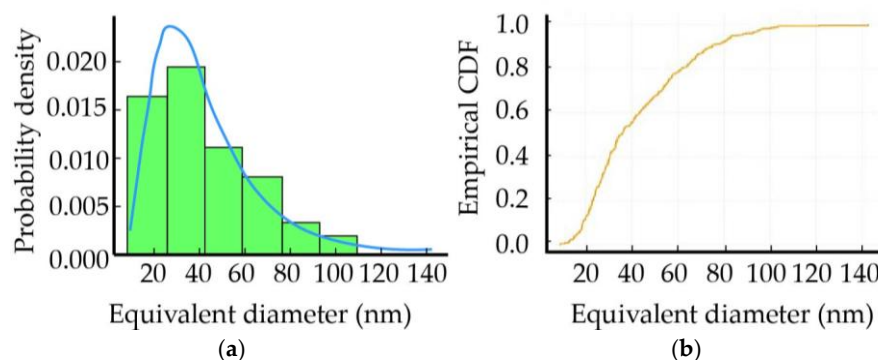


Figure 5. Log-normal approximation of the γ' -phase precipitate-size distribution (a). Empirical CDF (b).

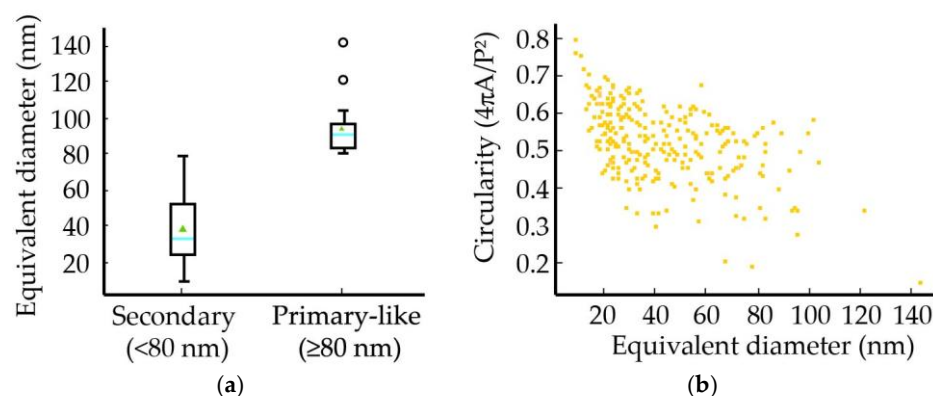
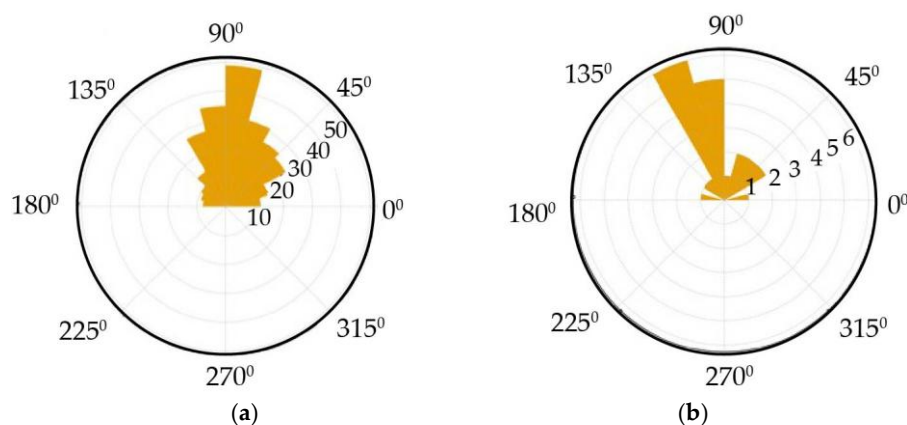
Microstructural classes and morphology. Box-plot classification by size classes is presented in Table 2.

Table 2. Box-plot classification by size classes.

γ' Precipitate Class	Size	Purpose
Secondary	<80 nm	Primary strengthening effect
Primary-like	≥ 80 nm	Indicator of partial coalescence

A negative correlation between diameter and circularity was also identified (Figure 6b), indicating particle deformation during coarsening and their transition toward a less coherent state, which may reduce creep resistance under long-term loading.

The pole figures (Figure 7a,b) show that the γ' -phase precipitates exhibit a certain texture orientation relative to the γ matrix. This is associated with the cubic coherency of the γ/γ' phases, typical of additive manufacturing, where a preferred growth direction exists along the build axis. Importantly, the smaller precipitates (<80 nm) exhibit more random orientations, indicating earlier nucleation and lower sensitivity to the matrix textured [7,86–88].

**Figure 6.** Classification of γ' -phase precipitate sizes (a). Morphology of the γ' -phase precipitates (b).**Figure 7.** Orientation-colored γ' -phase precipitates (<80 nm) (a). Orientation-colored γ' -phase precipitates (<80 nm) (b).

The observed characteristics of the γ/γ' microstructure are not only important for high-temperature strengthening but also directly influence hydrogen transport and trapping behavior. γ' precipitates, γ/γ' interfaces, and local microstructural heterogeneities may act as reversible or irreversible hydrogen trapping sites, affecting hydrogen distribution, stress localization, and the susceptibility of the alloy to hydrogen-assisted crack initiation. Therefore, the quantitative characterization of precipitate morphology and distribution provides an essential basis for understanding hydrogen-induced degradation mechanisms in Ni–Co superalloys.

4.2. Grad-CAM Heatmap Construction for the Ni–Co Superalloy Microstructure

Based on the SEM image of the Ni–Co γ/γ' superalloy microstructure (Figure 1a) and the methodology described in Section 3.4, a gradient-weighted class activation mapping (Grad-CAM) activation map was constructed and superimposed on the microstructure of the alloy (Figure 8). The resulting heatmap visualizes the spatial distribution of microstructural regions that have the greatest influence on the decision-making process of the convolutional neural network during microstructure classification and analysis.

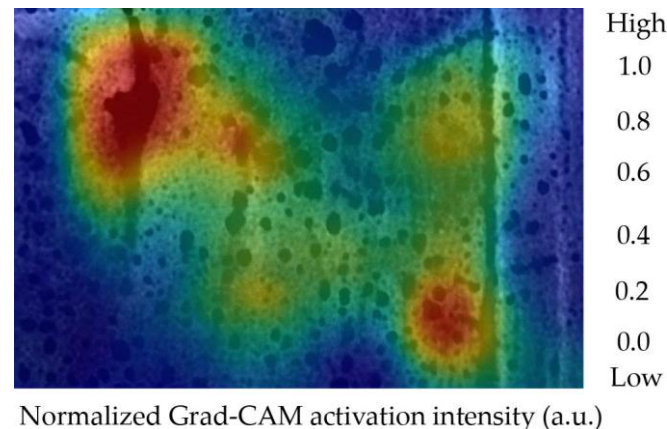


Figure 8. Grad-CAM activation map superimposed on the SEM microstructure of a Ni–Co γ/γ' superalloy.

Regions with high activation intensity (red and yellow areas) are predominantly localized in zones of increased microstructural heterogeneity, particularly near γ/γ' phase interfaces, local clusters of precipitates, pore-like features, and defect-rich regions. In contrast, areas characterized by a relatively homogeneous γ matrix with a uniform distribution of fine γ' precipitates exhibit low activation levels (blue and green regions).

The scale indicates the relative contribution of different microstructural regions to the neural network's decision, where low activation values correspond to blue regions and high activation values correspond to red regions.

This distribution indicates that the neural network identifies as most informative those microstructural features that are potentially associated with stress concentration, localization of plastic deformation, and hydrogen accumulation. Importantly, the Grad-CAM activation correlates well with regions where TEM-based morphometric analysis revealed γ' precipitate coarsening (>80 nm), partial loss of coherency, and an increased density of defects.

The obtained Grad-CAM results demonstrate that AI-assisted microstructural interpretation methods are capable of effectively identifying local regions prone to hydrogen-assisted degradation in Ni–Co γ/γ' superalloys. The pronounced activation of the neural network in the vicinity of γ/γ' interfaces, TCP phases, and defect clusters confirms their key role as effective hydrogen traps and preferential sites for microcrack initiation.

Thus, Grad-CAM not only enhances the interpretability of machine-learning-based analysis but also provides a physically meaningful link between digital diagnostics and the classical hydrogen embrittlement mechanisms (HELP/HEDE). This approach opens new opportunities for the early identification of degraded zones, optimization of heat-treatment strategies, and lifetime prediction of critical components in energy systems, including those manufactured using additive manufacturing technologies (AMTs). The application of ATM can help resolve overheating problems and enable the development of new blade repair technologies.

The regions highlighted by the Grad-CAM analysis correspond to microstructural features that are frequently reported as preferential sites for hydrogen accumulation and damage evolution. The concentration of neural-network activation near γ/γ' interfaces, coarsened precipitates, pores, and defect clusters suggests that these locations may play a critical role in the initiation of hydrogen-assisted microcracks.

4.3. Relationship Between Microstructure and Hydrogen-Induced Degradation Mechanisms in Ni–Co Superalloys

The microstructure of Ni–Co superalloys predominantly consists of a γ matrix with a high density of coherent γ' precipitates (spheres, cubes, aligned cubes, plates, short plates, doublets of short plates, octets of cubes, large plates, rafts, etc.). Intermetallic phases such as the η phase and the σ phase are also present in the structure. The main types of carbides in cast cobalt-based superalloys—MC, $M_{23}C_6$, M_7C_3 , and M_6C —are typically found both along grain boundaries and within the grains [89–92].

The heat treatment generally recommended for nickel-based superalloys is primarily intended to achieve a high-volume fraction and an improved size distribution of γ' precipitates, which provide optimal resistance to stress-induced rupture. In cast alloys with a high-volume fraction of γ/γ' eutectic, complete dissolution of the γ' phase through appropriate solution treatment is critically important [93–96].

The high melting temperature of nickel-based superalloys often allows refinement of the γ' microstructure by solution heat treatment followed by one or more aging stages [88]. The η phase is an intermetallic phase with an approximate composition of $Ni_3(Ti, Nb)$ and crystallizes in a hexagonal metastable structure (DO_{44}). (The DO_{44} structure—also written as $D0_{24}$ or $D0_{24}$ —is a structural type of intermetallic compound with a hexagonal mesoscale arrangement.)

It forms at high temperatures (approximately 750–900 °C), particularly during long-term aging or service exposure. In Ni–Cr–Co superalloys, at elevated Ti/Al ratios (>0.8) and temperatures around ~ 850 °C, the η phase precipitates after ~ 1000 h—more rapidly at higher Ti/Al ratios and temperatures [89]. A limited presence of the η phase can aid in controlling grain structure during forging (e.g., in A286 and IN706) by restricting grain growth [91]. The η phase may reduce the steady-state creep rate in Nimonic 263 [92]. An excessive amount of the η phase displaces the γ' strengthening phase, reducing tensile strength and impairing the balance of creep resistance at prolonged high temperatures. It also decreases room-temperature ductility and serves as a source of crack initiation along grain boundaries [93]. The η phase is a hexagonal $Ni_3(Ti, Nb)$ structure that forms at high temperatures in Ni–Co alloys. In moderate amounts, it may positively influence structural control or creep resistance; however, when present in excessive quantities, it degrades mechanical properties—reducing strength and ductility and accelerating crack propagation. The σ phase (sigma phase) is a brittle topologically close-packed (TCP) intermetallic phase that forms in Ni–Co and other superalloys.

It possesses a tetragonal crystal structure (space group $P4_2/mnm$) and is composed primarily of Cr, Co, Mo, W, Nb, and related elements [94]. Crystallography: the structure is tetragonal, space group $P4_2/mnm$, and the unit cell contains approximately 30 atoms occupying five nonequivalent positions. It was formed during prolonged aging or at high temperatures (>750 °C), especially in the presence of Co, Ni, Nb, and Al. Its presence has been confirmed in many HEA and Ni-based systems [94]. The effects of the σ phase include depletion of strengthening elements from the γ/γ' phases, embrittlement, reduced ductility, and decreased resistance to long-term load-induced failure or corrosion [95]. In the case of a Ni-based superalloy containing 13% and 35% Co, it has been shown that a higher Co content promotes the formation of the σ phase alongside γ' precipitates and carbides [95].

The stability of carbides at high temperatures is critical for the long-term performance of superalloys. Primary MC carbides are considered the most thermally stable; they remain solid even near the solidus temperatures of the alloy. In cobalt-based superalloys, the MC phase (e.g., NbC or TaC) serves as the primary strengthening constituent that persists at service temperatures of ~900–1000 °C and above [96]. Carbide phases in superalloys can exhibit different morphologies depending on their formation conditions. Primary MC carbides that form during solidification often possess coarse blocky or dendritic morphologies (the so-called “Chinese-script” carbides) and are typically located in the interdendritic regions of the cast structure [97]. Secondary $M_{23}C_6$ carbides, which precipitate in the solid state, typically exhibit a finer, more dispersed morphology—appearing as individual particles or thin films along grain or sub-grain boundaries. For example, in the René 108 alloy, nanoscale $M_{23}C_6$ layers with thicknesses of 5–15 nm were observed along the edges of primary carbides after aging [97]. Figures 9 and 10 show the general appearance of the microstructure of the Ni–Co alloys, illustrating the morphology of their structural constituents.

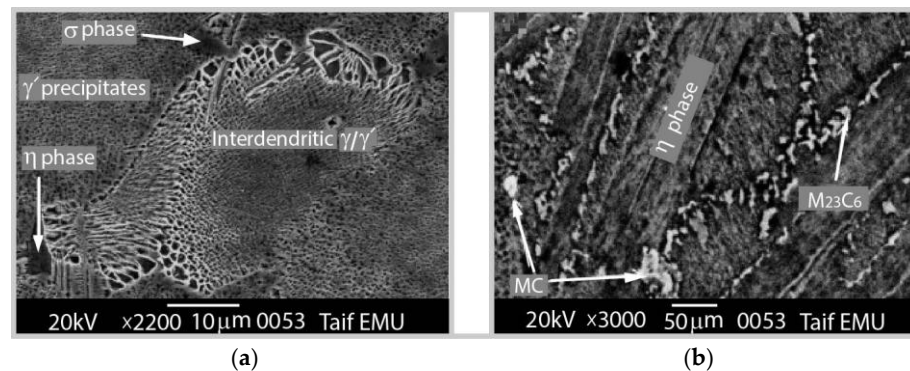


Figure 9. Microstructure of as-cast Ni-based superalloy (a). Aged microstructure at 845 °C [87,88] (b).

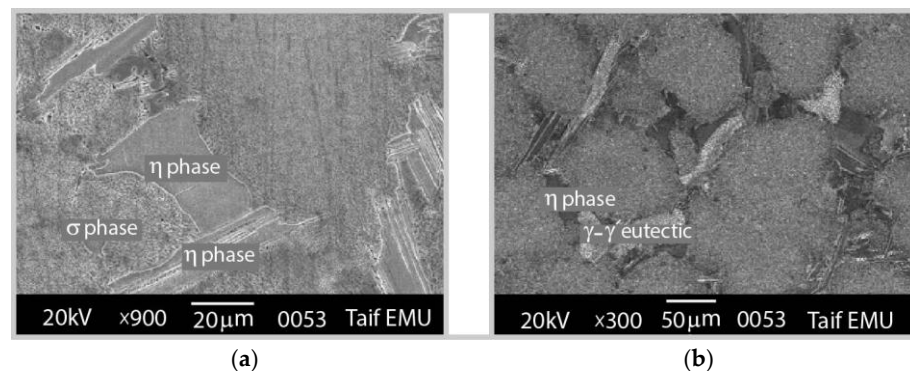


Figure 10. η-phase and σ-phase in as-cast microstructure (a). γ/γ' inter-dendritic and η-phase in cast microstructures [94] (b).

In addition to the η and σ phases, other intermetallic phases and carbides may also be present in Ni–Co superalloys [98,99].

In Figure 11, a conceptual consolidated scheme of the Ni–Co superalloy microstructure—before and after hydrogen charging—is presented. The main purpose of this scheme is to summarize the accumulated material to enable its further analysis and interpretation. A hydrogen-containing environment or hydrogen ingress may cause hydrogen embrittlement (HE) in superalloys—a reduction in ductility and strength resulting from the interaction of hydrogen with the microstructure. In Ni–Co superalloys, as in other high-strength alloys, hydrogen can accumulate at defects (dislocations, grain

boundaries, and phase particles) and promote premature crack initiation and growth (Table 3).

Table 3. Influence of intermetallic phases on the properties of Ni–Co superalloys.

Phase	Type	Stabilizing Elements	Impact on Properties.	Reference
γ' phase	L1 ₂ FCC (face-centered cubic)	Al, Ti, Nb, Ta	Increased high-temperature strength, precipitation strengthening; increased heat resistance, precipitation hardening; spherical shape.	[98,99]
γ'' phase (Ni ₃ Nb) coherent with FCC like γ'	BCT (body-centered tetragonal) DO ₂₂ (space group I4/mmm), coherent with matrix γ -Ni.	Stable up to 650 °C, with Ni ₃ Nb appear Ni ₃ V, Ni ₃ Mo, Ni ₃ Ta, Ni ₃ W	Ordered metastable intermetallic phase, Ni ₃ Nb; disk-shaped particles, with pronounced anisotropy along the c axis, which is responsible for the primary strengthening of superalloys. Lattice parameters γ'' : a $\approx$ 3.62–3.64 Å; c $\approx$ 7.41–7.49 Å.	[98–101]
η -phase (Ni ₃ Ti) non coherent	D0 ₁₉ TCP (topologically close packed)	Ti, Ta	Promotes embrittlement, impedes deformation. Forms slowly, which causes brittleness and makes deformation difficult. The absorbed hydrogen enhances the negative trends.	[100–103]
σ phase	TCP	Cr, Mo, W	Brittleness causes a reduction in ductility and fracture toughness, and a decrease in ductility and crack resistance. The absorbed hydrogen enhances the negative trends.	[104–107]
μ -phase, (Mu-phase)	TCP	Mo, W, Cr, Re	Reduced ductility. The absorbed hydrogen exacerbates the negative trends.	[107–109]
Laves phase	TCP	Nb, Ti, Mo	Embrittlement, failure. Absorbed hydrogen exacerbates negative trends.	[110–112]
β -phase	B2 orthorhombic	Ni–Al–Ti	High heat resistance.	[113–115]
R-phase	TCP	Re	Risk of cracking. The absorbed hydrogen exacerbates the negative trends.	[108]
P-phase	TCP	Re, Mo, W	A very fragile phase. The absorbed hydrogen exacerbates the negative trends.	[116,117]
δ -phase	Ni ₃ Nb	Nb	Borderline embrittlement. The absorbed hydrogen exacerbates the negative trends.	[118–121]
γ -phase (Ni)	HCC A1	Co, Cr, Fe, V, W, Ta	Strengthen the solid solution.	[120–123]

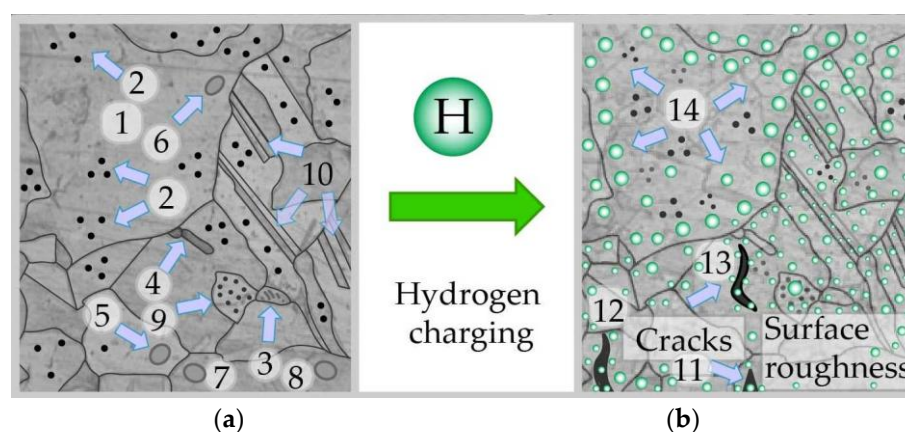


Figure 11. A schematic consolidated representation of the Ni–Co superalloy microstructure before hydrogen charging (a) and after hydrogen charging (b). Legend: 1— γ -matrix; 2— γ' precipitates; 3— σ phase; 4— η phase; 5—MC carbides; 6—M₆C carbides; 7—M₇C₃ carbides; 8—M₂₃C₆ carbides; 9—intermetallic phases; 10—twins; 11—crack on the component surface; 12—crack propagating into the component interior; 13—crack within the grain interior; 14—hydrogen.

Carbides play a dual role in this process: on the one hand, they may act as traps for hydrogen atoms, reducing their mobility; on the other hand, they may serve as stress concentrators and preferential crack-propagation paths, particularly when hydrogen accumulates at the carbide–matrix interface [124–127]. Carbides often settle at grain bounda-

ries [128–131], further contributing to brittle intergranular fracture under the action of hydrogen [132–135]. The interaction of carbides and other structural constituents with hydrogen therefore warrants special attention.

Despite the rapid development of casting, powder metallurgy, and 3D-printing technologies, as well as high hardness, thermal stability, and dispersion strengthening of Ni–Co superalloys, mechanical processing remains necessary during the final formation of the geometry of components, particularly in the manufacture of turbine blades and connecting elements, as well as during the repair and restoration of units that have undergone wear or deformation after long-term operation at high temperatures [136–139].

Hydrogen, entering the material either because of lubricating [140–143] and cooling fluids [144–147], or if necessary [148–151], repair work on equipment operated in a hydrogen-containing environment [152–155], significantly affects the machinability of steels and alloys [156–159]. Stress concentrations near intermetallic [160–163], carbides [164–167], and non-metallic inclusions [168–171] cause local accumulation of hydrogen and changes in the mechanisms of fracture during turning [172–175] and wear [4,176,177]. Novel hybrid additive manufacturing enables effective hydrogen trapping by producing layered structures similar to those found in two-dimensional anisotropic single crystals [169], where hydrogen becomes localized within the Van der Waals gaps, forming layer-by-layer barriers that impede hydrogen diffusion into the base material [168,169]. These phenomena have a common nature of aging from the perspective of the first physical principle (hydrogen with high parameters or the influence of high energy γ -irradiation on cast iron, steels, alloys, and chalcogenides in amorphous or single-crystal states) [172–174,178].

4.4. Additive Manufacturing and 3D-Printing Technologies for Energy Applications

At present, 3D printing [179–182] and related additive manufacturing [183–186] technologies are experiencing rapid and widespread implementation [187–191], supported by a strong technological background established through advances achieved in previous decades [192–194].

Ni–Co alloys are being extensively studied for processing by the selective laser melting (SLM) and directed energy deposition (DED) additive manufacturing routes [195–198].

In addition, a few studies have focused on the thermomechanical deformation mechanisms and high-temperature performance of these alloys in turbine disk prototype components [62,198–200].

The study by [201] demonstrated the feasibility of fabricating a γ/γ' -strengthened Co–Ni–Al–W superalloy by laser melting of a mixture of elemental powders, resulting in the formation of a high volume fraction of the γ' phase after appropriate heat treatment.

In study [97], the formation of a γ/γ' -strengthened Co–Ni–Al–W superalloy additionally alloyed with Cr and Ti and produced by laser melting of elemental powder mixtures, was investigated. It was shown that the addition of Cr and Ti increases the volume fraction of the γ' phase and enhances the oxidation resistance after heat treatment. At the same time, the incorporation of these elements reduces the γ/γ' lattice misfit, which leads to a decrease in creep resistance, a critical factor for high-temperature applications.

Mosallanejad et al. [66] provided a comprehensive review of in situ alloying in laser-based additive manufacturing processes, highlighting its potential for producing novel metallic alloys directly from elemental powder mixtures without prior powder atomization. The study underlined the necessity of precise control of laser processing parameters and appropriate post-processing heat treatments to achieve a uniform microstructure and stable mechanical properties.

The prospects for the application of 3D-printing technologies (LPBF/SLM, EBM, DED/WAAM) for Ni–Co superalloys in the energy sector are primarily associated with a

transition from ‘geometry-driven printing’ toward the controlled design of hierarchical microstructures and phase compositions (γ/γ' /carbides/TCP phases) tailored to specific high-temperature regimes and hydrogen-containing environments. Additive manufacturing enables local optimization of composition and properties (graded regions, topology optimization, internal cooling channels, repair and refurbishment of worn components); however, it also exacerbates the classical challenges of superalloys processed by AM, including hot cracking, porosity, anisotropy, residual stresses, and sensitivity to heat-treatment schedules. This necessitates an integrated process–structure–property approach and the development of standardized methodologies for defect control and micro-structural qualification [202].

For components operating in hydrogen-energy systems, a key issue is the integration of AM-based microstructural design with hydrogen resistance. Recent results indicate that realistic ‘hydrogen’ exposure scenarios (e.g., direct interaction with hydrogen flames) can lead to significant hydrogen uptake and non-trivial effects of oxide scales, while high-pressure H_2 environments are capable of drastically altering the fracture mechanisms of Ni-based superalloys [11,203].

Therefore, the most promising strategy appears to be a combined approach comprising (I) printing designed for γ' strengthening under controlled thermal cycling; (II) post-processing (HIP followed by aging) to close residual porosity and relieve internal stresses; (III) application of barrier/diffusion coatings and control of oxide scale formation; and (IV) qualification testing under relevant hydrogen environments. Taken together, these measures can accelerate the implementation of AM Ni–Co superalloys in turbomachinery, combustor components, and elements of hydrogen infrastructure.

4.5. Optimal Modern Chemical Composition of 3D-Printed Ni-Based Superalloys for High-Temperature Applications in Energy and Aviation

Various advanced Ni-containing steels [204–207] and superalloys [208–211], such as CM247LC [212–215], Hastelloy X [216–219], IN718, IN625, IN738, IN738LC [220–223], IN939, SRR99 [224–227], Haynes282, AD730 [228,229], ABD-900AM, ABD-850AM [230–233], and CMSX-4 [234,235], have been successfully produced using AM techniques [220,234,235]. Inconel 718 (UNS N07718) is one of the most widely used alloys for AM (selective laser melting, DED). This alloy is strengthened by precipitation of the γ'/γ'' phases ($Ni_3(Al, Ti, Nb)$), which provide high-temperature strength and resistance to hydrogen embrittlement. Inconel 625 (UNS N06625) is used for components requiring strength at high temperatures combined with high corrosion resistance. In contrast to Inconel 718, Inconel 625 is strengthened primarily by solid-solution strengthening due to the Nb/Mo relations. Haynes® 282 was developed for 3D-printing technology and exhibits the best heat-resistance characteristics and weldability.

Inconel 718 (UNS N07718)—suitable materials for AM, selective laser melting, DED, etc. Typical chemical composition: Ni: 50–55%; Cr: 17–21%; Fe: balance; Nb + Ta: 4.75–5.50%; Mo: 2.80–3.30%; Ti: 0.65–1.15%; Al: 0.20–0.80%; Co: up to 1%; Mn, Si: $\leq 0.35\%$; C: $\leq 0.08\%$; B, P, S: $\leq 0.01\%$. This alloy is strengthened by precipitation of the γ'/γ'' phase ($Ni_3(Al, Ti, Nb)$) and exhibits high heat resistance and creep resistance at high temperatures. Inconel 625 (UNS N06625) is also a suitable material for AM due to its high heat resistance and resistance to high-temperature corrosion. Typical chemical composition (mas. %): Ni: $\approx 58\%$; Cr: 20–23%; Mo: 8–10%; Nb + Ta: 3.15–4.15%; Fe: $\approx 5\%$; Co, Mn, Si, Al, Ti: $\leq 0.01\%$; C: $\approx 0.1\%$. Not strengthened in the same manner as 718 alloy; the primary strengthening procedure is solid-solution strengthening by Nb/Mo.

Haynes® 282 is more suitable for 3D printing with the best characteristics of heat resistance and weldability. Cr: oxidation-resistant; Co, Mo, Al, Ti, and Nb: strengthening elements. Haynes 282 (N07208) belongs to the Ni–Cr–Co type of alloy, which consists of

Mo and W (9–11%), Nb (4–5%), Al (2.5–3.5%), Ti (1.5–2.5%), Cr (19–21%), Co (8–10%), Fe (up to 1%), C and B—for high strength at high temperatures, thermal stability and weldability for application in gas turbines. Typical chemical composition (mas. %): Ni: balance (~51–55%); Cr: 19.0–21.0%; Co: 8.0–10.0%; Mo: 9.0–10.0%; W: 9.0–10.0%; Nb: 4.0–4.5%; Al: 2.5–3.5%; Ti: 1.5–2.5%; C: 0.05–0.08%; B: 0.005–0.01%; Fe: ≤1.0%; Mn: ≤0.5%; Si: ≤0.5%; Cu: ≤0.1%; P: ≤0.015%; S: ≤0.010%. The roles of main alloying elements: Cr: corrosion resistance and strengthening; Co, Mo, W: strengthening at high temperatures, increasing of heat resistance; Nb, Al, Ti: as a component of γ' gamma prime phase; C, B: has improved grain boundary strength and stability. This composition has made the Haynes 282 suitable for badges production, combustor chamber and other high temperatures components applications in aerospace and energetics. The main differences between Inconel 718 and Haynes 282: Inconel 718 typically exhibits higher low-temperature strength and better room-temperature mechanical properties; while Haynes 282 provides temperature strength and creep resistance, making it more suitable for applications above 650 degrees C (1200 degrees F). Haynes 282 also provides better weldability and machinability, but its higher strain rate sensitivity and higher cost (due to higher cobalt and molybdenum content) can make it more difficult to machine.

For medium- and high-temperature structural components that require excellent long-term creep resistance, thermal stability above approximately 650 degrees C (1200 degrees F), and good weldability, Haynes 282 is generally the best choice. For applications that require extremely high room temperature, temperature and medium-temperature yield/strength, wide availability of various forging forms and many years of experience in the oil and gas and aerospace industries, Inconel 718 remains the preferred choice. Ultimately, the selection of the appropriate alloy depends on the operating temperature, the required creep time, the manufacturing process (intensive welding or forging) and cost and supply constraints.

Haynes 282 (UNS N07208) and Inconel 718 (UNS N07718) are nickel-based superalloys designed specifically for high-temperature applications, but their designs meet different performance requirements. Haynes 282 is a phase-strengthened wrought superalloy designed to balance high creep resistance at high temperatures with good weldability and machinability. It is suitable for components with hot gas passage, advanced steam and supercritical carbon dioxide (s-CO₂) cycles, and other applications requiring long creep life at temperatures above 650 degrees. Inconel 718 is a nickel-chromium-iron dispersion alloy (primary hardener: γ'' Ni₃Nb phase) that exhibits excellent strength from low temperatures to approximately 650 degrees. Its combination of machinability, high yield/aging strength, and mature supply chain has led to its widespread use in the aerospace, oil and gas, and tooling industries. Inconel 718 appears to be more difficult to process in energetic flow machine than Haynes 282, mainly due to its higher strength and greater strain rate sensitivity. Inconel 718 has corrosion resistance is dependent on the environment; it performs better than Haynes 282 in some alkaline environments, but is more susceptible to corrosion in acidic environments (e.g., HCl). Haynes 282 provides better corrosion resistance than Inconel 718 in some acidic environments due to the formation of a denser passivating film. Alloys CM-247/RR1000/MAR-M247 are also used for heat resistance enhancement, especially in turbine blades and other hot areas of engines. Typical chemical composition: Ni: >50–60%; Cr: 12–16%; Co: 8–12%; Mo, W, Ta, Re (for hardening at high temperatures); Al + Ti: ~6–8% for γ' -precipitation (example of high-performance superalloys adapted for AM). γ' -strengthening Ni-based superalloys such as IN718, IN738LC [95,97], DZ125L [98], and Rene 104 [99] are also used during metal AM processes.

Powder characteristics: AM uses gas-dynamically or plasma-sprayed powders with a strictly controlled composition that match the composition of the corresponding alloy

grades. Impurity control: even minor impurities (O, N) in powders can seriously increase defect formation and affect the mechanical properties of parts, especially for Inconel 718-based alloys. Composition and microstructure analysis: Additively manufactured products may have an uneven distribution of alloying elements due to the rapid solidification and thermal cycling of the process, requiring further heat treatment to optimize properties.

Inconel 718 (Ni, Cr, Nb, Mo, Ti, Al)— γ'/γ'' precipitation; high strength up to ~ 700 °C Inconel 625 (Ni, Cr, Mo, Nb)—strength + corrosion resistance Haynes 282/analogue (Ni, Cr, Co, Mo, Al, and Ti) better weldability and AM properties CM-247/RR1000 etc. (Ni, Cr, Co, Mo, W, Ta, Re, Al, and Ti) very high heat resistance for turbine blades.

EP-741P, available only in powder form, and VZHL-14, available both in powder and cast forms, were compared with forged EP-915, which has a similar composition (it is an analogue of EP-666). 741 alloys are stronger but more sensitive to HE. In AM, the manufacturing process and subsequent HT are important. Inconel 625 (UNS N06625) is used for components which need strength at high temperatures combined with high corrosion resistance. In contrast to Inconel 718, its primary strengthening mechanism is solid-solution strengthening due to Nb/Mo relations. Haynes 282 has been specially created for 3D-printing technology and exhibits the best heat-resistance characteristics and weldability.

5. Conclusions

1. A comprehensive multiscale analysis of hydrogen-induced nanoscale degradation and crack initiation in γ/γ' -strengthened Ni–Co superalloys, which are promising materials for energy and turbomachinery applications, was carried out. According to the results of transmission electron microscopy and quantitative morphometry, a dense and homogeneous distribution of coherent γ' precipitates with characteristic sizes in the range of 40–120 nm, which are described by the log law and have a predominantly spherical shape, was established.
2. Statistical analysis revealed a correlation between particle size, aspect ratio, and roundness, indicating a partial loss of coherence and the onset of coagulation for γ' precipitates larger than ~ 80 nm. The presence of TCP phases (η , σ , μ , Laves) and various types of carbides at grain boundaries and within grains increases microstructural heterogeneity and creates highly efficient hydrogen traps, promoting decohesion at interfacial boundaries and microcrack initiation.
3. The application of the Grad-CAM method to SEM images allowed us to visualize the microstructural regions most sensitive to hydrogen-assisted damage and to emphasize the decisive role of γ/γ' interfaces, TCP phases, and defect clusters in crack initiation.
4. It is established that hydrogen-induced degradation in Ni–Co γ/γ' superalloys is determined by the interconnected system “microstructure–hydrogen–stress state”, in which the morphology of nanoscale precipitates, intermetallic phases, and defects caused by additive manufacturing control hydrogen capture, the localization of plastic deformation, and the transition from micro-void formation to microcrack growth. The results obtained create a physically sound basis for optimizing the chemical composition, heat treatment regimes, and 3D-printing parameters, as well as for developing digital and AI-oriented models for predicting the lifetime of critical components of hydrogen-cooled generators and gas hydrogen turbines.
5. Additive manufacturing technologies (LPBF/SLM, EBM, DED/WAAM) show strong potential for producing and repairing Ni–Co superalloy components for energy applications, including turbine disks, combustor parts, and elements of hydrogen infrastructure. AM processing introduces specific challenges such as hot cracking, residual porosity, chemical inhomogeneity, anisotropy, and residual stresses, which re-

quire optimized alloy design, processing parameters, and post-treatments to support predictive models for the durability of critical components in hydrogen-energy and high-temperature power-generation systems with increased hydrogen safety.

6. For hydrogen-energy systems, the integration of AM-based microstructural design with hydrogen resistance is critical, since recent studies have demonstrated significant hydrogen uptake, complex roles of oxide scales, and changes in fracture mechanisms in Ni-based superalloys under hydrogen flames and high-pressure H₂. An integrated strategy combining γ' -oriented printing, HIP and aging, and the use of diffusion-barrier coatings with controlled oxide layers appears to be the most promising route to ensure long-term durability.

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Conflicts of Interest: The authors declare no conflicts of interest.

Nomenclature and Abbreviations

AM	additive manufacturing
GTEs	gas-turbine engines
GTUs	gas-turbine units
LCL	lubricating cooling liquids
C _H	hydrogen concentration
ppm	parts per millions
TA	turbo aggregate (turbine + turbogenerator)
HCTG	hydrogen-cooled turbogenerator
SLM	selective laser melting
°C	degree Celsius
TEM	transmission electron microscopy
BD	build direction
CDF	cumulative distribution function
APS	atmospheric plasma spraying
DED	directed energy deposition
EBM	electron beam melting
EBSD	electron backscatter diffraction
EP-741P	nickel-based superalloy, powder variant
EP-742	nickel-based superalloy, deformable variant
GTT	gas-turbine technology
HAZ	heat affected zone
HE	hydrogen embrittlement
HEA	high-entropy alloy
HEDE	hydrogen-enhanced decohesion
HELP	hydrogen-enhanced localized plasticity
HESIV	hydrogen-enhanced strain-induced vacancy
HIP	hot isostatic pressing
H ₂	hydrogen
HV	Vickers hardness
IoT	Internet of Things
ISO	International Organization for Standardization
K _{IC}	fracture toughness
K _{th}	threshold stress intensity factor

LCL	lubricating cooling liquids
LPBF	laser powder bed fusion
MC	metal carbide
MQL	minimum quantity lubrication
SEM	scanning electron microscopy
SLM	selective laser melting
TEM	transmission electron microscopy
TCP	topologically close-packed phases
TCH	total combustion hydrogen analyzer (LECO)
γ	gamma-matrix phase
γ'	gamma-prime strengthening phase (Ni ₃ (Al, Ti, Co))
η	eta phase (Ni ₃ (Ti, Nb))
σ	sigma phase (Cr, Mo, W rich TCP phase)
AI	artificial intelligence

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